



TITLE:

Morphology of Organometallic Precipitates by Electron Microscope

AUTHOR(S):

Ishibashi, Masayoshi; Suito, Eiji; Takiyama,
Kazuyoshi; Sekido, Eiichi

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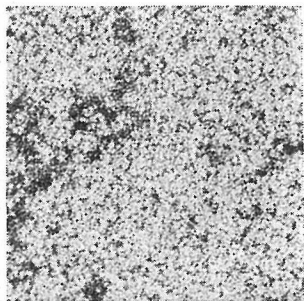


Photo 3. $\text{Fe}(\text{OH})_3$.
 $\times 15,000$

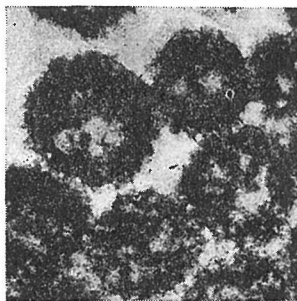


Photo 4. $\text{Fe}(\text{OH})_3$.
 $\times 8,000$

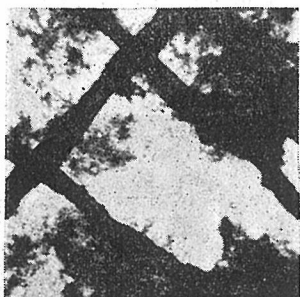


Photo 5. $\text{Fe}(\text{OH})_3$.
 $\times 5,000$

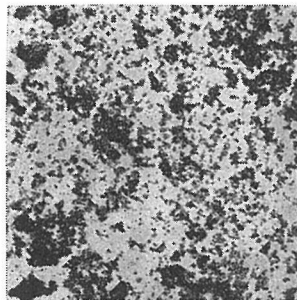


Photo 6. $\text{Al}(\text{OH})_3$.
 $\times 15,000$

4. Morphology of Organometallic Precipitates by Electron Microscope

Masayoshi ISHIBASHI, Eiji SUITO,
Kazuyoshi TAKIYAMA and Eiichi SEKIDO

(Ishibashi and Suito Laboratories)

The shape and size of the precipitate are important for the analytical chemistry. Recently the organometallic compounds have attracted attention for quantitative analysis. And the authors studied organometallic compounds the precipitates of Ni-, Pb-dimethylglyoxime, Cu-, Al-, Pb-, Mg- oxinate and Cu-, Al-cupferrate were observed by electron microscope.

Precipitates were prepared by mixing of necessary reagents for each sample. Ni-dimethylglyoximes were precipitated from NiSO_4 solution of 2.0, 1.0, 0.5, 10^{-1} , 10^{-2} , 10^{-3} , 10^{-4} and 10^{-5} M. But the other precipitates were prepared at the usual condition for the quantitative analysis.

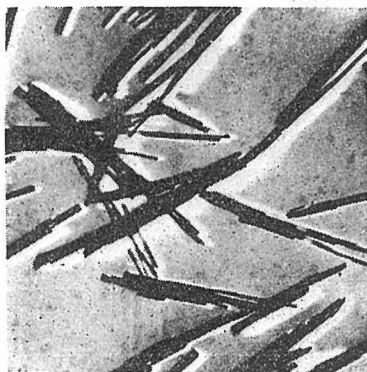


Photo 1. Ni-dimethylglyoxime
from 0.1 M NiSO_4 .
(Cr-shadowing) $\times 5,000$

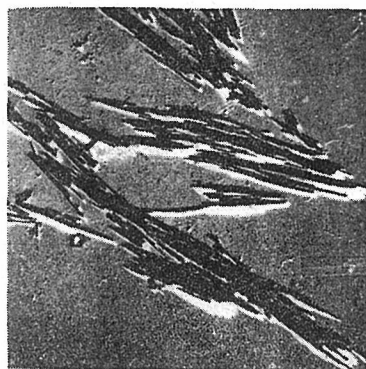


Photo 2. Ni-dimethylglyoxime
from $10^{-3} M$ NiSO_4 .
(Cr-shadowing) $\times 5,000$

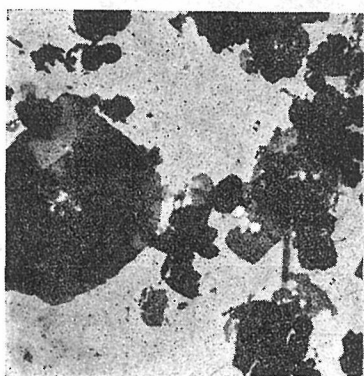


Photo 3. Ni-dimethylglyoxime.
 $\times 6,000$

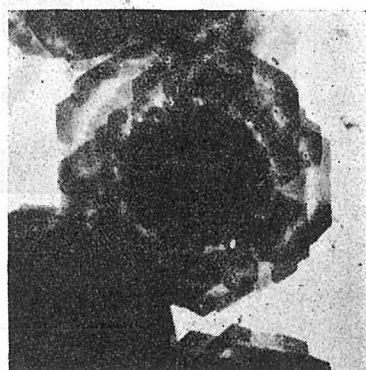


Photo 4. Pb-oxinate.
 $\times 6,000$

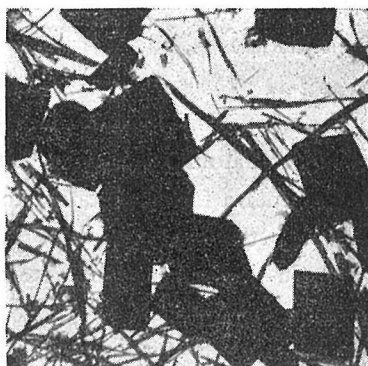


Photo 5. Cu-oxinate.
Coexisting needle crystal is
oxine. $\times 6,000$

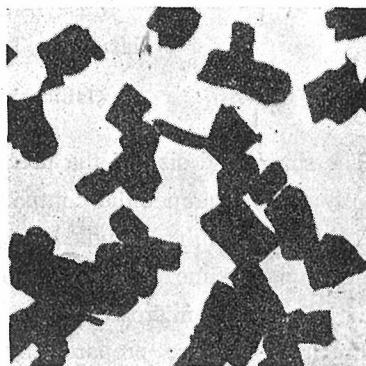


Photo 6. Cu-cupferrate.
 $\times 10,000$

The condition of precipitations and the size and shape of the precipitated particles are shown in Table 1.

Table 1. Size and shape of precipitates.

Sample	Condition of precipitation	Size (μ)	Shape
Ni-dimethylglyoxime	ammoniacal	1~3*	slender rod
Pb-dimethylglyoxime	ammoniacal	2~1	thin hexagon
Cu-oxinate	HAc, NaAc buffer	3~4	rectangle
Cu-oxinate	ammoniacal	2~1	rectangle
Al-oxinate	HAc, NaAc buffer	2~4	rectangle
Mg-oxinate	NH ₄ OH, NH ₄ Cl	5×1**	rod
Pb-oxinate	ammoniacal	1~1.5	thin hexagon
Cu-cupferrate	HAc, NaAc buffer	0.5~1	tetragon
Al-cupferrate	HAc, NaAc buffer	—	—

* The width of the particles is almost constant, 0.1 μ .

** The length is 5 μ and the width is 1 μ .

About the relation of the concentration of NiSO₄ solution to the shape and size of Ni-dimethylglyoxime, it was observed that each shape of the precipitate was slender rod, and the width of the rod was almost constant (0.1 μ), but the length became smaller little by little with the decrease of the concentration (3 μ ~1 μ), but at 10⁻⁴ M the size became a little larger (2 μ). (Photo 1 and 2). When 0.01 M ferrous ion was added in 10⁻⁴ M NiSO₄ solution, the shape of Ni-dimethylglyoxime precipitate was not rod but a slender needle.

It seems that the shape of the particles precipitated with the organic reagent will depend on the nature of metal ion, that is, the shape of the precipitates of Pb-dimethylglyoxime and Pb-oxinate was thin hexagonal plate (Photo 3 and 4), and that of Cu-oxinate and Cu-cupferrate was rectangular plate (Photo 5 and 6), but the conclusive results can not be given unless many experiments are performed. The precipitates of Mg-, Al-oxinate and Al-cupferrate have a tendency to coagulate. The precipitates of Pb-dimethylglyoxime and Pb-oxinate tend to aggregate in a large hexagonal plate.

Almost the organometallic compounds observed here were changed by strong electron bombardment. And the change occurs not only in their shapes, but also in their structures. This fact can be recognized by the change of the electron diffraction pattern. For example, the pattern of Pb-oxinate was changed after long and strong electron bombardment.